

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s): Ralph Bauer et al.

Title: SURFACE COATING SOLUTION

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## DECLARATION UNDER 37 C.F.R. §1.132

Sir, I hereby declare and state:

1. I am a joint inventor of the subject matter presently claimed in the above-identified patent application.
2. I received my undergraduate degree in Metallurgical and Materials Engineering from Middle East Technical University, Ankara, Turkey in 1997, received my M.S. degree in Ceramic Engineering from Alfred University in 1998, and received my Ph.D. in Materials Science and Engineering from Pennsylvania State University in 2001.
3. For over 8 years, I have been involved in the research and development of alumina powders. Since 2001, I have been employed by Saint-Gobain Ceramics & Plastics, Inc. (or its predecessor companies), during which time I have primarily been engaged in research and development of alumina powders.
4. I have reviewed have reviewed Elsik et al. (US 5,550,180), Bugosh (US 2,915,475), and Gernon et al. (US 2006/0106129, hereinafter “Gernon ‘129”).
5. Background. The claimed invention is directed to a surface coating solution including a surface coating base and boehmite particles provided in the surface coating base in an amount of 0.1 wt% to 20.0 wt%. The boehmite particles are mainly anisotropically shaped particles

having an aspect ratio of at least 3:1. The surface coating solution has flow and leveling of at least 6 and a sag resistance of at least 7 mils.

The claimed sag resistance and flow and leveling properties and other claimed thixotropic properties, including viscosity recovery, were found to result from particular process features for forming surface coating solutions, coupled with use of anisotropic boehmite particles, particularly those boehmite particles formed through a seeded process. Such notable process features include activation of the boehmite particles prior to incorporating the boehmite particles into a grind solution.

In contrast, the cited reference Elsik does not disclose a formula having the flow and leveling and sag resistance recited in the claims.

#### 6. Examples 1 and 3 of Elsik.

Testing was performed on latex formulations based on Example 1 and Example 3 of Elsik, with the exception that Rhoplex AC-264 is used in place of the UCAR 516 latex emulsions. The resulting formulations did not exhibit a flow and leveling of at least 6 and a sag resistance of at least 7 mils.

In review of the raw materials employed in the preparation of the latex formulations of Elsik, it was found that the UCAR 516 latex emulsion was discontinued and is not commercially available. The UCAR 516 latex emulsion was an acrylic-vinyl chloride terpolymer. According to former Union Carbide personnel, the UCAR 516 latex emulsion product was discontinued due to health and safety concerns. After an extensive internet search and follow-up with numerous latex emulsion suppliers, a vinyl chloride terpolymer as low in molecular weight and glass transition temperature as the UCAR 516 was not found.

Thus, in order to reproduce the examples of Elsik, another type of latex emulsion that is commonly utilized in the formulation and production of interior and exterior paints was selected. The emulsion selected for the formulations is Rhoplex AC-264, a 100% acrylic emulsion supplied by Rohm & Haas that has been widely used in the manufacturing of paints since the early 1980's and thus has a great deal of recognition among formulators in the architectural paint arts.

Table 1 illustrates the pertinent properties of the Rhoplex AC-264 and UCAR 516.

TABLE 1. PROPERTIES OF LATEX EMULSIONS

Property	Rhoplex AC-264	UCAR 516
Appearance	Milky White Liquid	Milky White Liquid
Weight Solids	60.0%	55.0%
pH	9.2	9.0
Specific Gravity	1.06	1.09
Weight per Gallon	8.9 lbs/gal	9.1 lbs/gal
Viscosity	500 cps	250 cps

In preparing the formulations with Rhoplex AC-264, the level of latex emulsion in the formulation was adjusted so that the revised formula with Rhoplex AC-264 had the same solids content as the UCAR 516 based formulations of Example 1 and Example 3 of Elsik. Since the weight solids of Rhoplex AC-264 is 5 % greater than that of the UCAR 516, the UCAR 516 was replaced on a solids basis. The difference in weight, after adjustment based on solids, was made by adding water.

In addition, the level of Texanol, the coalescing agent for the latex, is adjusted from 8.4% to 7.5 % to accommodate the lower coalescent demand of the Rhoplex AC-264 in Example 1 and from 7.5 % to 6.0% in Example 3.

Details of the formulations are illustrated in Table 2 and Table 3. Table 2 illustrates the formulations for Interior Flat Wall Paints prepared in accordance with Example 1 of Elsik with the above-noted exceptions. Table 3 illustrates the formulations for Exterior House Paints prepared in accordance with Example 3 of Elsik with the above-noted exceptions.

TABLE 2. Formulations Prepared in Accordance with Example 1 of Elsik

Material	Alumina (P2) (lbs)	HEC + Alumina (P2) (lbs)
DI Water	100.00	170.00
Dispersal P2 Boehmite Alumina	5.76	4.14
Natrosol 250HUR Modified HEC	-----	2.76
** Mix on a High Speed Disperser for 20 minutes @ 2000 RPM, then add: **		
Nuosept 145 Biocide	1.84	2.64
Propylene Glycol	21.12	30.35
Foamaster NDW Defoamer	1.36	1.95
Triton X-100 Surfactant	2.96	4.25
Tamol 731 Pigment Dispersant	5.84	8.39
AMP-95	2.96	4.25
Ti-Pure R-931 Titanium Dioxide	106.08	152.44
Calcium Carbonate	87.60	125.89
ASP-NC Aluminum Silicate	51.12	73.46
** Mix on a High Speed Disperser for 20 minutes @ 2000 RPM, then add: **		
Foamaster NDW Defoamer	1.36	1.95
Rhoplex AC-264 Acrylic Latex	291.20	418.49
DI Water	26.48	38.04
Texanol	13.21	18.98
DI Water	79.92	89.85
	798.81	1147.83

TABLE 3. Formulations Prepared in Accordance with Example 3 of Elsik

Material	HEC (lbs)	HEC + Alumina (lbs)
DI Water	242.2	242.2
Dispersal P2 Boehmite Alumina	-----	2.6
Cellosolve QP-15000	3.5	1.8
** Mix on a High Speed Disperser for 20 minutes @ 2000 RPM, then add: **		
DYK VP-155	9.1	9.1
Triton N-101	2.0	2.0
Colloid 640	6.9	0.9
KTPP	1.0	1.0
Nuosept 145 Biocide	2.0	2.0
Nuocide 960	7.0	7.0
Propylene Glycol	28.0	28.0
Ti-Pure R-900 Titanium Dioxide	250.00	250.0
Mimex 7	150.00	150.0
Optiwhite Calcined Kaolin Clay	50.0	50.0
** Mix on a High Speed Disperser for 20 minutes @ 2000 RPM, then add: **		
Rhoplex AC-264 Acrylic Latex	378.2	378.2
Ammonium Hydroxide	1.8	1.8
Texanol	13.6	13.6
Colloid 640	2.6	2.6
DI Water	34.4	34.4
	1176.3	1177.2

As indicated in the above formulations, in both Example 1 and Example 3 of Elsik, the formulations were prepared to assure that the alumina (Disperal P2) and the HEC were dispersed for 20 minutes at 2000 RPM to assure dispersion prior to pigmentation. After the pigment was added, the grind base was dispersed for another 20 minutes at 2000 RPM. After preparation, the samples were allowed to stand at room temperature for 24 hours to let the formulations equilibrate. At this point, the testing was initiated utilizing the test methods listed in Table 4 below.

TABLE 4. Testing Methods

<u>Property</u>	<u>Test Method</u>
Viscosity	
Stormer	ASTM D562
Brookfield	ASTM D2196
SAG Resistance	ASTM D4400
Flow and Leveling	ASTM D2801
Dry Times	ASTM D1640 (circular dry time recorder)
Specular Gloss	ASTM D523

The properties of the latex formulations are illustrated in Table 5 and Table 6. Table 5 illustrates the properties of Interior Flat Wall Paints prepared in accordance with Example 1 of Elsik with the above-noted exceptions. Table 6 illustrates the properties of Exterior House Paints prepared in accordance with Example 3 of Elsik with the above-noted exceptions.

TABLE 5. Properties of Formulations According to Example 1 of Elsik

Property	Alumina (P2)	HEC + Alumina (P2)
Stormer Viscosity (K.U.)	63 K.U.	85 K.U.
Brookfield Viscosity (cps)		
20 RPM	990	3085
50 RPM	554	1830
100 RPM	400	950
Recovery Time	9 minutes	2.5 minutes
SAG Resistance	< 1 mil	5 mils
Flow and Leveling	6	5
Dry Times		
Set-to-Touch	21 minutes	21 minutes
Surface Dry	1.2 hours	1.2 hours
Specular Gloss		
60 Degree	12.4	10.2
20 Degree	2.1	2.0

The formulation prepared in accordance with Example 1 of Elsik including alumina (P2) alone exhibited a significantly lower sag resistance and slightly greater flow and leveling than did the formulation prepared with the Disperal P2 alumina and the HEC. Neither sample exhibited both flow and leveling of at least 6 and sag resistance of at least 7 mils.

TABLE 6. Properties of Formulations in Accordance with Example 3 of Elsik

Property	HEC	HEC + Alumina (P2)
Stormer Viscosity (K.U.)	95 K.U.	76 K.U.
Brookfield Viscosity (cps)		
20 RPM	5220	2350
50 RPM	3200	1256
100 RPM	2140	845
Recovery Time	55 seconds	2.0 minutes
SAG Resistance	12+ mils	8 mils
Flow and Leveling	1	2
Dry Times		
Set-to-Touch	18 minutes	21 minutes
Surface Dry	1.2 hours	1.2 hours
Specular Gloss		
60 Degree	4.2	4.5
20 Degree	1.5	1.5

The formulations prepared in accordance with the Example 3 of Elsik including HEC alone exhibited significantly greater sag resistance than the formulation prepared with the Disperal P2 alumina and the HEC. Neither exhibited flow and leveling of at least 6.

Improvements in flow and leveling often come with a sacrifice to sag resistance and vice-versa. A desirable thickener achieves a balance between the sag resistance and flow and leveling. While the above samples fail to achieve such a balance, examples provided in the

present specification achieve such a balance. In particular, examples in the present specification exhibit flow and leveling of at least 6 and sag resistance of at least 7 mils.

7. The prior art.

As demonstrated above, samples formulated in accordance with Examples 1 and 3 of Elsik that include alumina particulate do not have flow and leveling of at least 6 and sag resistance of at least 7 mils. As explained below, Elsik fails to disclose a latex formulation that has the claimed properties and is free of associative thickener. Bugosh fails to disclose activation of boehmite and fails to provide details regarding paint formulations. Gernon '129 fails to teach or suggest use of boehmite thickeners.

Elsik is directed to latex compositions including, as a rheology modifier, a boehmite alumina. (Elsik, Abstract). In Examples 1-4, Elsik discloses use of Disperal Sol P2 in amounts of around 0.22 wt% to 0.7 wt% in an acrylic-vinyl chloride modified latex. In Example 5, Elsik provides the properties of other boehmite alumina, such as Disperal and Catapal D.

As demonstrated above and in a prior Declaration signed February 2, 2009, samples prepared using various commercially available boehmite particulates in the amounts identified by Elsik did not produce latex paint having flow and leveling properties of at least 6 and having a Leneta sag resistance in the range of 7 mils to 12 mils. Instead, the Disperal P2 samples, even when activated, exhibit poor flow and leveling, and the Disperal and Catapal D samples exhibit poor Leneta sag resistance.

Turning to the other cited references, Bugosh is directed to fibrous aluminum monohydrate particles. Bugosh further discloses that fibrous boehmite can be used as reinforcing filler in making plastic films, coatings, paints, adhesives, or other plastic articles. The fibrous boehmite may be mixed with aqueous dispersions of polymers. (Bugosh, col. 29, ll. 1-21). Bugosh is silent regarding composition of the coatings and paints and is silent regarding characteristics of the coatings and paints, such as flow and leveling, sag resistance, and set-to-touch dry time characteristics. While, as disclosed by Bugosh, it may have been known to incorporate boehmite into coatings, paints, and adhesives, Bugosh is silent regarding activating

the boehmite particulate and is silent regarding the process for forming aqueous dispersions of polymers.

Gernon '129 is directed to latex paint formulations that contain N-n-butyl ethanolamine (BAE) as a neutralizing agent. Gernon '129 discloses a flat interior paint that includes a Polyphobe 102 rheology modifier and other coatings that include RHOPLEX® or Acrysol® rheology modifiers. The flat interior paint reportedly exhibits a leveling of 8. Gernon '129 does not disclose the use of a boehmite rheology modifier.

Further, replacing a thickener of Gernon with an anisotropic boehmite would influence the properties of the modified Gernon paint, particularly leveling, pH, and viscosity. Accordingly, replacing the thickener of Gernon would not necessarily provide a latex paint having desirable sag resistance and flow and leveling, or even desirable pH, or viscosity.

In contrast to the cited references, my co-inventors and I have discovered that the present anisotropic boehmite particles advantageously produce surface coatings having desirable characteristics, such as desirable flow and leveling, sag resistance, set-to-touch dry time, and shear viscosity recovery. As explained above, not all paint formulations that include boehmite exhibit the claimed combination of properties.

8. Summary. Among the features disclosed in the present application, the above-mentioned activation of anisotropic boehmite particles contributes to successful formation of a surface coating solution having the claimed properties. Nevertheless, as illustrated by the examples provided, not all formulations that include boehmite thickener have the claimed sag resistance and flow and leveling properties. In particular, samples prepared in accordance with Example 1 and Example 3 of Elsik do not exhibit flow and leveling of at least 6 and sag resistance of at least 7 mils.

The foregoing innovations were created by my co-inventors and me through extensive research and development, and are at least partly the result of empirical studies on the notable engineering hurdles associated with the formation of surface coating solutions.

9. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like, so made, are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

08/17/09  
Date

  
Doruk O. Yener, Ph.D.